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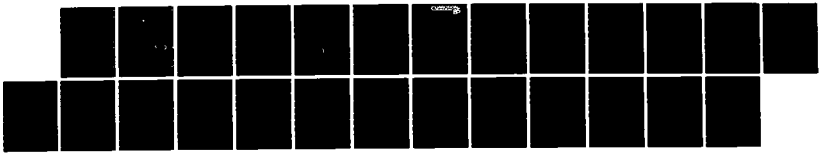

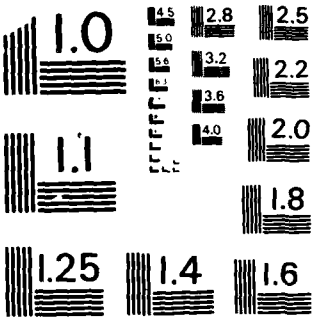

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THE EFFECT OF BAKING AND STRESS ON THE HYDROGEN CONTENT OF CADMIUM PLATED HIGH STRENGTH STEELS

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THE EFFECT OF BAKING AND STRESS ON THE HYDROGEN CONTENT OF CADMIUM PLATED HIGH STRENGTH STEELS

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ABSTRACT

Variations in the diffusible hydrogen concentration of cadmium plated AISI 4340 and 300M steels due to baking and stress are reported. Long baking times of greater than 100 hours at 190 C are needed to remove all the hydrogen from bright cadmium plated specimens, and around 40 hours for dull cadmium plated ones. Very short baking not only does not remove hydrogen, but actually pumps it in. Baking for 96 hours is insufficient to remove hydrogen embrittlement from sharp, double notched 300M tensile specimens having a notched tensile strength of 1800 MPa. It is shown that there is no difference in the hydrogen concentration whether baking immediately follows plating or there is a delay. Also, the hydrogen concentration does not change with time for cadmium plated specimens standing for one month at room temperature. Stressing, on the other hand, causes an increase in the hydrogen, presumably by allowing it to enter from the plate due to lattice deformation.

INTRODUCTION

Electrodeposited cadmium is widely used as a sacrificial coating to protect low alloy, high strength steels from corrosion, especially in marine environments.[1] It is generally recognized, however, that hydrogen produced during this plating can diffuse to areas of high stress causing embrittlement and possibly leading to catastrophic failure of a structural part, such as an aircraft landing gear.[2] Because of this, plated parts are baked to lower the hydrogen concentration and thus reduce the possibility of failure.

Specifications for baking are often ill-defined, sometimes calling for as little as 1-5 hours at a wide range of temperatures or stating that the time and temperature should be determined for the material and intended use.[3] Thus, in the aircraft industry, cadmium plated parts are baked for 24 hours at 375 F (190 C) for the highest strength steels. This has been shown to be insufficient to eliminate embrittlement from a part,

especially when it is covered with a bright cadmium plate electrodeposited from conventional cyanide baths.[4,5]

Common practice and specifications are usually to "bake as soon as possible after plating."[3] This is done to prevent cracking due to residual stresses. Also, it is often assumed that early baking is necessary because the hydrogen is more easily removed at this stage. Baking immediately may be good housekeeping as it assures continuity of the procedure so that the baking is not omitted; however, whether or not there is any benefit from a hydrogen viewpoint has not been established.

Because of the difficulties with hydrogen embrittlement when using bright cadmium, low embrittlement plating baths are often used. These produce duller, more porous plates which lose hydrogen more easily. The dull cadmium is not as attractive or protective as the bright, however. Therefore, the latter is still preferred for many uses.

Traditionally, mechanical testing has been the only way to determine the effectiveness of the baking procedure. Since the development of the barnacle electrode method,[5,6] however, it is now possible to measure diffusible (embrittling) hydrogen and thus to monitor baking efficiencies. The barnacle electrode technique is based on the electrochemical hydrogen permeation method. If hydrogen is in a part which is made the anode in an electrochemical cell, the hydrogen is oxidized as it diffuses to the surface. The oxidation current can then be related to the hydrogen content of the part. Correlations with mechanical testing are, of course, still necessary to determine safe levels of hydrogen for the material and use.

Stressing produces a lattice expansion which causes hydrogen to move along the stress gradient and, over a period of time, to build up to high levels.[7] Because of this time dependence, measurement of the time to failure in a sustained (constant) load test is used to predict susceptibility of a material to hydrogen embrittlement. It is therefore important that methods are found to correlate the diffusible hydrogen content and susceptibility to embrittlement, especially of cadmium plated high strength steels. Hence, the present work to study the effects of baking and stress on the hydrogen concentration and delayed failure was undertaken.

In recent years, statistical methods have been used, not only in analyzing experimental data but, as importantly, in designing the experiment so as to get the most information from the least number of specimens.[8,9] Due to the complexity of the plating/baking procedures, statistical design concepts were therefore used here.

EXPERIMENTAL

Materials

The specimens used in the screening experiments were 38 mm square, 1.0 mm thick pieces of AISI 4340 steel heat treated to 260-280 ksi (1800-1900 MPa) strength level. The remainder of the studies were conducted with 300M steel heat treated to 280-300 ksi (1900-2100 MPa) strength level. The double notched 300M tensile specimens were 6 in. long and 0.063 in. thick (150 and 1.60 mm), and had a geometrical stress concentration factor, k_t, of 5.6. The notched tensile strength (NTS) was 260 ksi (1800 MPa). The 300M coupons were 75 x 35 mm, either 2.0 or 6.0 mm thick. All specimens were finished by surface grinding.

Procedures

The bright cadmium electroplate was applied from a cyanide bath, containing brighteners, at a current density of 25 A/ft^2 (25 mA/cm^2); the dull from a cyanide bath, containing carbonate, at a current density of 60 A/ft^2 (60 mA/cm^2). All plates were 0.0005 in. (13 um) thick, and all specimens were baked at 375 F (190 C). The cadmium was removed for hydrogen measurements using ammonium nitrate solution, followed by light abrading with Scotchbrite.

All hydrogen measurements were made using the barnacle electrode method. Details of the equipment and procedures are given elsewhere.[5] The 30-minute extraction current densities were used to give the relative diffusible hydrogen concentrations. Measurements on stressed specimens were made as soon as possible after failure, at least 25 mm away from the fracture surface.

The tensile specimens were loaded in constant load stress rupture machines for the time to failure determinations.

In all experiments, randomness was used where necessary, such as in the plating bath, oven and loading frames to ensure statistical reliability.

Statistical Experimental Design

Screening experiments were run to determine the effect of five variables on the diffusible hydrogen concentration of both bright and dull cadmium plated 4340 steel coupons. The 12-run Plackett-Burman experimental design[8,10] is shown in Table 1. Each column has the limits for a particular variable given, the plus or minus standing for high/low, #1/#2, etc. Column Y is for the response, the measured hydrogen concentration. The variables and their limits are given in Table 2. As the choice of assigning variables to columns is arbitrary,[8,10] the five variables were assigned to the first five columns of Table 1. This automatically defined which of the two levels (e.g., baking for 8 or 48 hours) was to be used for each specimen. Thus, for column X_1 (Table 1), trials (specimens) 1, 2, 4, 5, 6 and 10 are all pluses which means plating batch one (Table 2) for the bright cadmium experiment and batch two for the dull. Likewise, specimens 2, 3, 4, 8, 10 and 11 were baked for 48 and 20 hours for the bright and dull, respectively. The remaining six columns (X_6- X_{11}) have a random distribution of pluses and minuses which were used to determine the experimental error.

Statistical Analysis

The effect of each variable was determined by comparing the hydrogen concentration for the specimens in which that variable was at one level to those at the other. This was done by adding the Y-values for all the pluses in the column, subtracting the Y's of the minuses, and dividing by six (the number of pluses or minuses) to get the factor effect for the variable. This factor effect was then compared to the minimum significant factor effect, [MIN], to determine its statistical significance.

The [MIN] was calculated using the unassigned columns. First, the unassigned factor effect, UFE, for each column was calculated in the same way as above. This was then used to determine the significant factor effect, S_{FE}, which is an estimate of the experimental error,

$$S_{FE} = \sqrt{\frac{\sum_{i=1}^{q} UFE_i^2}{q}} \qquad (1)$$

where q is the number of degrees of freedom (the number of unassigned columns). Then

$$[MIN] = t \cdot S_{FE} \qquad (2)$$

where t was obtained from the t-distribution table[8] using q=6.

To determine the importance of the variables, their factor effects were then compared to the [MIN]. If the absolute value of the factor effect is larger than the [MIN], then the variable is statistically significant.[8]

RESULTS AND DISCUSSION

Screening Experiments

The hydrogen concentrations for the 12 specimens, as measured by the barnacle electrode method, are given in Table 3 for both the bright and dull cadmium experiments; also given is column X_3 from Table 1 and a sample calculation for the factor effect for baking. The average difference in the hydrogen concentrations for the

specimens baked for the short and long times was calculated by taking the absolute difference of the sums and dividing by six. Thus, for the bright cadmium experiment, the sum for specimens 2, 3, 4, 8, 10 and 11 is 3.26; for specimens 1, 5, 6, 7, 9 and 12 is 5.36. The difference is 2.10; and the factor effect is 0.35. This factor effect was then compared to the minimum significant factor effect, [MIN], to determine its importance. The latter is given in Table 4 for both the 90 and 95% confidence levels. The factor effects for the five variables are also given in Table 4. One can see that for the bright cadmium, only the baking time is significant, whereas with the dull cadmium, the batch is also significant (starred values). The reason for this is probably that small changes in concentration of the bath ingredients, especially carbonate, can have a significant effect on the nature of the deposit from a dull cadmium bath. In this experiment, the two batches were plated one week apart. In the case of the bright cadmium, the batch could become a significant factor for more extreme changes in the plating conditions. In all subsequent tests in which two or more plating batches were used, the plating was always done on the same day by the same technician. Also, specimens were distributed so as to eliminate a possible batch effect.

The factor effects for the other variables were shown to be statistically non-significant, Table 4. Thus, baking immediately, or a day after plating will not affect the hydrogen concentration. (This does not, of course, preclude the possibility that materials under stress might be damaged if baking is delayed.) The delay in measuring the hydrogen also was found to be non-significant up to 690 hours (approximately one month) at room temperature and moderate humidities. This means that delayed failure testing vs diffusible hydrogen measurements can be made with confidence that the hydrogen concentration will not change with time. It also means that interlaboratory, or other testing, involving delayed hydrogen measurements can be done with no need to coordinate the times for measurements. Extremely long times at very high humidities could alter these conclusions.

Because of the concern voiced by many plating people that a delay between plating and baking could be important, it was decided to run a check experiment with just the two variables: the baking time and the delay before baking. Bright cadmium plated specimens were baked for 3 and 72 hours, with dealys before baking of ½ and 24 hours. Duplicate specimens were run for the four combinations of high and low values as shown in Table 5. Also given in Table 5 are the diffusible hydrogen concentrations as determined by the barnacle electrode method. As can be seen, the measurements on the specimens baked for the short time are in excellent agreement, as are those baked for the long time, regardless of the delay time; i.e., trials 1 and 2 agree with each other, as do 3 and 4. It has thus been shown, unequivocally, that there is no effect on the hydrogen concentration whether or not the baking is done "as soon as possible" after plating.

Effect of Baking

As we have seen, baking (and to some extent, the plating batch) is the only variable which was found to affect the final diffusible hydrogen concentration. The effect of baking time on the removal of hydrogen from cadmium plated 4340 steel is illustrated in Figure 1. The curve for the dull cadmium plated specimens is the best fit for the 12 points from the five-variable screening experiment; that for the bright cadmium plated is from the two-variable experiment. For the latter, data from an intermediate baking time of 37.5 hours (not shown in Table 5) was also used. Extrapolations of the curves show that complete bake-out would be obtained at times well beyond any normal baking procedures. It would take over 100 hours for specimens having the bright cadmium coating, and even 40 hours for the dull. The "no hydrogen" level is the measurement obtained with plated and stripped specimens which were allowed to degas in a desiccator. The very low concentrations of hydrogen measured for the

longer baking times are much too small to be calculated quantitatively, but are well under 0.1 ppm.[5]

Effect of Stress

Next, constant load experiments were run with 300M steel tensile specimens. Because the screening experiment showed that the hydrogen concentration does not change over a month, it was thought that the hydrogen could be measured on the failed specimens, and that this would give the same value as if it had been measured before loading. In early trials, however, this did not seem to be the case. There was some indication that the hydrogen concentration may have been affected by the stressing. Even though it is well known that stressing produces diffusion of hydrogen to regions of high stress, it is not apparent that the bulk hydrogen concentration is affected to any measurable extent upon stressing. Therefore, the effect of stress on the hydrogen concentration was investigated by measuring the hydrogen in control (unstressed) specimens and comparing it to that in stressed and failed ones.

A preliminary two-variable, two-level, constant load experiment was conducted to determine appropriate baking times and loads for the 300M steel. Duplicate specimens were run for each bake/load condition. The results of the time to failure tests are given in Table 6. The failure times were generally very short, with very little discrimination in times for the different bake/load conditions. Thus all specimens failed within seven hours except for the 48 hr/25% condition. The expected trend is obvious, however; that is, the shortest times to failure are at the lower left (short bake/high load) and increase for longer baking time or lower load.

Due to the short failure times in the preliminary experiment, longer baking times were selected for the main experiment in order to reduce the hydrogen levels and extend the times to failure. Triplicate specimens were run for each bake/load condition. The results of this experiment are given in Table 7. Again the trends are apparent. Thus, specimens baked for 72 hours and stressed at 75% NTS failed in shorter average time than those in the 96 hr/75% condition, and the former in shorter time than those stressed at 25% NTS. Comparing Tables 6 and 7, it can be seen that the largest spreads in times to failure are with the lowest stresses (25% NTS). This can be explained as follows. When stressing, hydrogen moves from the bulk of the specimen to regions of high stress, due to lattice deformations.[7] When loading at low stress levels, however, these lattice deformations are slight. Therefore, small variations in hydrogen concentration, or metallurgical and mechanical factors are probably very important in affecting the life of the specimen. Contrast this with the effects for the 96 hour bake and 90% load. Here the lattice deformation is great, thus causing the hydrogen to build up to damaging levels in relatively short times, even though the starting hydrogen concentration was low after the long baking time.

To determine the effect of stress on the hydrogen, the concentration of diffusible hydrogen was determined on the stressed specimens after failure, as well as on the unstressed ones. The results are given in Table 8. Looking at the hydrogen concentrations for the control specimens, Table 8, it can be seen that over 100 hours of baking at 190 C is needed to remove all the diffusible hydrogen. This is in agreement with the screening experiments using 4340 steel. The very low level of hydrogen after baking for 96 hours is still embrittling, however, as seen in Table 7. It should be noted that the background current density level for the screening experiment is different from that in the stressing experiment. This value must be determined for each set of specimens, as it depends on both the material and the surface finish.

Comparisons of the hydrogen concentrations for the stressed and unstressed specimens (the latter combined according to baking time without regard to load), Table 8, show that stressing does, indeed, affect the hydrogen

concentration. For all baking times, the stressed specimens had higher hydrogen levels than the controls. Further, a two-factor computer analysis of variance was run using the Statistical Package for the Social Sciences (SPSS).[11] It was found that there was a significant difference in the hydrogen concentration for the stressed versus the unstressed specimens at the 95% confidence level.

One would normally expect a loss of hydrogen through the fracture surface during cracking, a process that often takes several hours to complete. Therefore the increase in hydrogen can only be explained by the addition from a source, such as from the cadmium plate (or possibly from traps in the steel). Stressing causes a lattice expansion in both the steel and the cadmium. The hydrogen in the cadmium could then move into the steel, and along the stress and hydrogen concentration gradients to the notch area, giving both the buildup to cause failure, and an increase in the bulk hydrogen concentration.

Effect of Short Baking

Since even long baking times (up to 96 hours) were shown to be insufficient to remove hydrogen, it was decided to investigate the effect of short baking as specified,[3] to see if there was any benefit at all. Rectangular coupons (76 x 38 mm) of 300M steel were plated with bright cadmium, and baked for different times. Table 9 gives hydrogen concentrations for 6 mm thick specimens baked for times up to 24 hours. It can be seen that there is no removal of hydrogen in these times. In fact, there is some indication that hydrogen may even be increased during the first few hours. To check this out, a second experiment was run using both 2 and 6 mm thick coupons. The hydrogen concentrations are given in Table 10. It is clear that baking for only an hour or two causes an increase in diffusible hydrogen, the time depending on the thickness of the specimen. Using t-test calculations[12] to compare the hydrogen concentration for the unbaked specimens against those baked for one and two hours, for the 2 mm and 6 mm thick specimens, respectively, significant differences at the 95% confidence level were found. Thus, it has been shown that very short baking (1-5 hours) does not remove any hydrogen and may, in fact, cause an increase.

CONCLUSIONS

Several experiments were run to study the effects of baking and stress on the diffusible hydrogen concentration of cadmium plated high strength steels.

Screening experiments were performed to determine the statistical significance of various parameters on the hydrogen content of bright and dull cadmium plated, and baked, 4340 steel. It was shown that the baking time was essentially the only significant variable influencing the final hydrogen concentration as determined by the barnacle electrode method. However, variations between plating batches were found to be significant for the dull cadmium plated material.

A delay of 24 hours in baking after plating had no effect on the final hydrogen concentration. Also, the hydrogen concentration was not changed if the plated specimens were held for a month at relative humidities up to 50% after baking.

It was found that the hydrogen measurements decreased exponentially with the baking time, and that at least 100 hours (at 190 C) were needed to bake out all the detectable hydrogen from 4340 and 300M steel specimens plated with 0.015 mm of bright cadmium, whereas 40 hours were required with dull cadmium.

It also was shown that baking up to 24 hours had no effect on removing hydrogen from bright cadmium plated material, and that hydrogen actually increased during the first few hours of baking.

In constant load experiments, it was found that embrittlement still existed after 96 hours of baking at 190 C for bright cadmium plated 300M

steel having a NTS of 1800 MPa and a sharp notch, and that the hydrogen content of the steel increased after stressing. It is proposed that hydrogen enters the base metal from the plate during stressing. Therefore, the possibility of hydrogen embrittlement of very high strength steels plated with bright cadmium is always present, given the right combination of stress and time.

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TABLE 1 - 12-Run Plackett-Burman Experimental Design

Trial	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	Y
1	+	+	-	+	+	+	-	-	-	+	-	
2	+	-	+	+	+	-	-	-	+	-	+	
3	-	+	+	+	-	-	-	+	-	+	+	
4	+	+	+	-	-	-	+	-	+	+	-	
5	+	+	-	-	-	+	-	+	+	-	+	
6	+	-	-	-	+	-	+	+	-	+	+	
7	-	-	-	+	-	+	+	-	+	+	+	
8	-	-	+	-	+	+	-	+	+	+	-	
9	-	+	-	+	+	-	+	+	+	-	-	
10	+	-	+	+	-	+	+	+	-	-	-	
11	-	+	+	-	+	+	+	-	-	-	+	
12	-	-	-	-	-	-	-	-	-	-	-	
Sum +												
Sum -												
Difference												
Effect												

TABLE 2 - Variables and Limits for Plackett-Burman Experiments

Column*	Variable	Limits			
		Bright Cadmium		Dull Cadmium	
		-	+	-	+
X_1	Plating batch	2	1	1	2
X_2	Time between plating and baking, h	1	24	1	24
X_3	Baking time, h	8	48	8	20
X_4	Time between baking and measuring, h	2	690	2	690
X_5	Relative humidity**, %	0	35	0	50

* Column corresponds to Table 1.

** 0, over Drierite; 35, average ambient; 50, over saturated $Na_2Cr_2O_7$.

TABLE 3 - Hydrogen Concentrations and Sample Calculations for Plackett-Burman Screening Experiments

Trial	Bake*	Hydrogen Concentration,** μA/cm² Bright Cadmium	Dull Cadmium
1	-	1.15	0.68
2	+	.34	.27
3	+	.74	.30
4	+	.54	.50
5	-	.97	.56
6	-	.97	.65
7	-	.78	.42
8	+	.33	.34
9	-	.58	.36
10	+	.60	.36
11	+	.71	.30
12	-	.91	.43
Sum +		3.26	2.07
Sum -		5.36	3.10
Difference***		2.10	1.03
Factor Effect (÷ 6)		0.35	0.17

* See column X_3, Table 1.
** Background level, 0.22 μA/cm².
*** Absolute value.

TABLE 4 - Factor Effects and Minimum Significant Factor Effects, [MIN], for the Plackett-Burman Experiments

Variable	Factor Effect Bright Cadmium	Dull Cadmium
X_1 Batch	0.087	0.145*
X_2 Delay before baking	.127	.038
X_3 Baking time	.350*	.172*
X_4 Delay after baking	.040	.065
X_5 Humidity	.077	.005
S_{FE} (experimental error)	.119	.048
[MIN] 95% confidence level, t=2.45	.291	.118
[MIN] 90% confidence level, t=1.94	.230	.093

* Significant factor effects

TABLE 5 - Two variable, Two-level Experimental Design and Results

Trial	Baking Time, h	Delay Time, h	Hydrogen Concentration, $\mu A/cm^2$	
				Avg
1	3	½	0.88	
			1.26	1.07
2	3	24	1.08	
			1.02	1.05
3	72	½	.26	
			.31	.28
4	72	24	.28	
			.31	.30

* Background level, 0.22 $\mu A/cm^2$

TABLE 6 - Effects of Baking and Stress on Failure Time of 300M Steel
(Preliminary Experiment)

Load* % NTS	Times to Failure, h	
	Baking Times, h	
	24	48
85	4.7	41.5
	5.7	177.0
75	0.0	3.1
	1.0	6.8

*Notched tensile strength, 1800 MPa.

TABLE 7 - Effects of Baking and Stress on Failure Time of 300M Steel

Load*, %NTS	Times to Failure, h		
	Baking Times, h		
	48	72	96
25		16	
		104	
		>500	
50	4		
	8		
	14		
75		0	13
		5	35
		51	46
90			30
			32
			89

* Notched tensile strength, 1800 MPa.

TABLE 8 - Effects of Baking and Stress on Hydrogen Concentration of 300M Steel

Baking Time, h	Stress	No. Specimens	Hydrogen Concentration,* $\mu A/cm^2$	
			Avg.	Std. Dev.
0	No	4	0.60	0.076
48	No	4	.45	.086
	Yes	3	.50	.118
72	No	4	.42	.073
	Yes	6	.52	.070
96	No	4	.38	.037
	Yes	6	.44	.056

* Background level, 0.35 $\mu A/cm^2$.

TABLE 9 - The Effect of Short Baking Times on the Hydrogen Concentration of Cadmium Plated 300M Steel

Baking Time, h	Hydrogen Concentration, $\mu A/cm^2$	
	Avg.*	Std. Dev.
0	0.38	0.026
2	.42	.063
4	.43	.025
8	.42	.033
14	.44	.049
24	.43	.073

* Three 6 mm thick specimens each.

TABLE 10 - The Effect of Very Short Baking Times on the Hydrogen Concentration of Cadmium Plated 300M Steel

Baking Time, h	Hydrogen Concentration, $\mu A/cm^2$			
	2 mm Thick		6 mm Thick	
	Avg.*	Std. Dev.	Avg.*	Std. Dev.
0	0.44	0.023	0.43	0.031
1	.54	.082	.43	.063
2	.50	.041	.53	.029
4	.45	.015	.52	.039

* Four specimens each.

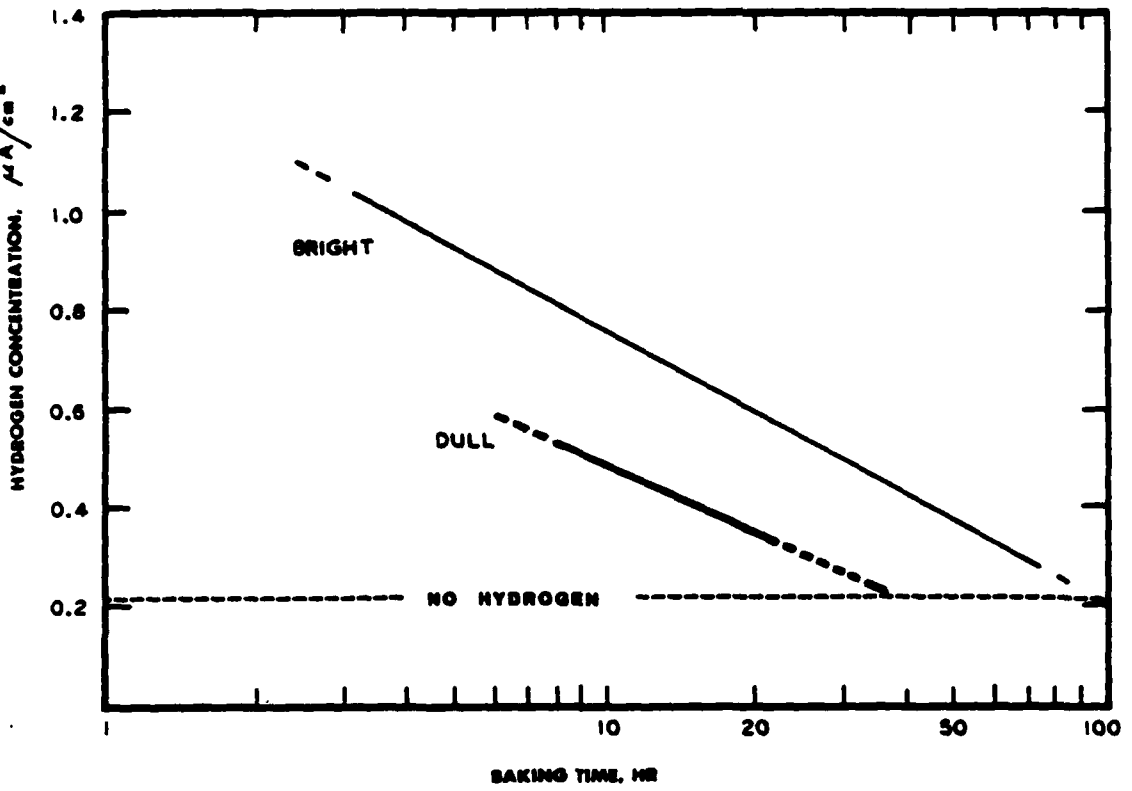

FIGURE 1 - Efficiency of baking treatment for removal of hydrogen from bright and dull cadmium plated AISI 4340 steel. Plating thickness, 15 μm. Baked at 190 C.

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